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A concrete performance test for delayed ettringite formation: Part I optimisation

Alexandre Pavoine *, Loïc Divet, Stéphane Fenouillet

Laboratoire Central des Ponts et Chassées. 58 Bd Lefebvre, 75732 Paris Cedex 15 — France

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Abstract

Delayed ettringite formation (DEF) is a rare internal swelling reaction of concrete that, in a wet environment, may considerably reduce the durability of a structure or a member that has been prefabricated in a factory. As a result of a large number of studies, the main causes of this problem have been established and this paper proposes a test method to predict the susceptibility of a concrete to DEF and provide long-term protection against this hazard. After an examination of the feasibility of a test based on wetting and drying cycles, the results of an optimisation study based on a factorial experimental plan are presented. The test is in four stages: concrete manufacture, a heat treatment that simulates steam curing in a factory or the heating that occurs in mass cast in-situ concrete, wetting and drying cycles and the monitoring of the longitudinal expansion of specimens that are immersed in water. Optimisation essentially concerns the last two stages and resulted in the decision to apply two drying cycles (38 °C at a relative humidity of approximately 30%) and wetting cycles (in water at 20 °C) followed by permanent immersion in water (at 20 °C). This test is applied to concrete that has undergone early age heating. It provides a means of evaluating the potential reactivity of "mix design–heating cycle" pairs as realistically as possible within a reasonable period of time.

Keywords: Delayed ettringite formation; Expansion; Sulfate attack

1. Introduction

Delayed ettringite formation (DEF) is the term used to describe a physicochemical process that may lead to the premature deterioration of a concrete member. DEF is an internal swelling reaction of the concrete that occurs merely in the presence of water without any external ingress of sulfate from the environment in contact with the concrete. This concrete swelling reaction is influenced by a large number of factors that play a role in its development. The main parameters involved in DEF are as follows:

Temperature. A temperature rise considerably modifies the chemical equilibrium during cement hydration as well as the texture of the material [1–3]. It is recognized that a high temperature is a necessary condition for delayed ettringite formation. For this reason, the problem can only occur in mass concrete due to self heating or in concrete that has undergone heat treatment. The normally stated critical temperature is around 70 °C. To limit risks in the long term, current recommendations propose, among other things, a maximum temperature of 60–70 °C for heat curing [4–9].

Water. In situ, appraisals have shown that the presence of water in contact with the concrete is a necessary condition for delayed ettringite formation. The role of high relative humidity around the concrete has also been confirmed by laboratory studies [10–13].

Alkali in the concrete. This is known to affect the solubility of ettringite [14]. As the solubility of ettringite also varies with temperature, there is a strong interaction between these two parameters during the DEF process [15–17].

Initial cracking of the concrete. This probably has an effect on the kinetics and extent of expansion. However, the topic is controversial and the available theoretical investigations [18] and experimental studies [19] do not allow us to reach a conclusion with regard to the importance of this parameter.

Sulfate in the clinker. Recent changes in cement manufacturing processes have changed the amount and nature of the

* Corresponding author.
E-mail address: alexandre.pavoine@lcpc.fr (A. Pavoine).

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sulfates in the clinker. A considerable number of kinetic and thermodynamic studies have been conducted to evaluate the role of these sulfates in the swelling process. Laboratory tests have shown that these sulfates only account for a small proportion of the total amount of sulfate in the cement, that their proportion in relation to aluminates favours the formation of monosulphoaluminate and that their solubility kinetics are such that they may be dissolved during binder hydration [20]. It is accepted that these sulfates cannot be responsible for long-term expansion without heating of the material. However, expansion tests have demonstrated that the sulfates present in the clinker, as alkali sulfates, modify the amplitude and the kinetics of expansion of the material in the event of considerable early age heating [19]. Nevertheless, there is no evidence that the sulfates in the clinker have a different effect from those added.

Sulfates and aluminates in the cement. Ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12}$ $26H_2O$) is a trisulfo calcium aluminate hydrate. Sulfates and aluminates therefore play an important role in the reaction mechanism. Studies have been concerned with the equilibria between sulfates in solution, hydrated sulfates and C–S–H during hydration and over a period of time. During hydration, these equilibria are considerably modified by heating as a result of the effect of the temperature on the texture of the C–S–H, changes in the domains of thermodynamics stability of hydrates and variations in the products of solubility. This subject has been widely discussed in the literature that deals with the reaction mechanisms [1,2,15,20–22]. The role of the aluminates in the cement has frequently been studied and C_3A limits or criteria which relate to both aluminates and sulfates have been put forward. Nevertheless, in view of the complexity of the phenomenon, these criteria taken individually are not always relevant.

Additives. Adding substances that modify the physical characteristics that influence the durability of concrete (pozzolanic reactions, porosity) seems to reduce the potential risk of a given concrete expansion [23]. However, few studies have explored this topic. Recent results with regard to air-entraining agents describe the impact of included air bubbles which can act as expansion vessels without, in principle, reducing the material's frost susceptibility [24,25].

Although progress has been considerable, controversial points, or areas where data is lacking, exist and these limit our overall understanding of DEF. However, it is of great interest to try to develop performance tests [16] to meet the requirements of project owners and contractors.

Some countries have already introduced restrictions to reduce the risk of DEF [4–9]. In the case of France, the official recommendation is to assess the reactivity of a concrete using a performance test when certain temperature (from 65 °C to 85 °C) and concrete composition criteria (low C_3A, SO_3 and Na_2O_{equi}) cannot be met. With regard to the management of existing structures, a technical guide has recently been published in France in which the use of this type of test is also recommended for structures or members in which the reaction would threaten either the safety of users or the durability of the structure [26].

The literature proposes several tests to evaluate the reactivity of concrete in relation to DEF [12,27,28]. Until now, these tests have been performed on small specimens (of cement paste or mortar) and have not been shown to be generally applicable because very high temperatures are applied on one or more occasions. More recently, Petrov [25] has shown that a discriminant test can be achieved by varying the temperature and the relative humidity during a performance test. However, when these tests were conducted on mortar, a large difference was noted between the amplitude of expansion of the control specimens (without cycles) and the test specimens.

A performance test must be a representative of the reality. It must also be possible to carry out the test within a reasonable length of time. To meet these constraints, we have applied a three-stage approach:

1. Verification of the feasibility of a concrete test involving the application of wetting and drying cycles and expansion measurement [29].
2. Optimisation of the test based on the application of a factorial experimental plan described in Table 1.
3. Validation of the test by applying it to concrete with known behaviour with regard to DEF [30].

2. Experimental description

2.1. The proposed test

During the feasibility study, it was observed that internal sulfate reaction may cause damage to concrete [29]. The similarity between the results obtained on specimens stored in water, known as control specimens (without cycles), and specimens subjected to wetting and drying cycles was checked. When concretes were susceptible to DEF, expansion was measured on both specimens without cycles and those subjected to drying and wetting cycles. Non-reactive concretes did not expand when the drying cycles were carried out at 38 °C. These first results led to the proposal of a four-phase performance test. The first phase consists of the manufacture of concrete

Table 1
Scope of study for optimizing the performance test

Reference letter	Parameter	Levels
A	Concrete curing time before application of drying and wetting cycles	5 h; 3 days; 14 days
B	Time kept in a water-saturated enclosure (relative humidity close to 100%)	0 day; 3 days; 7 days
C	Duration of a wetting and drying cycle	7 days; 14 days; 28 days;
D	Number of cycles before permanent immersion of concrete specimen	1 cycle; 2 cycles; 3 cycles
E	Drying temperature	38 °C; 60 °C
F	Solution specimens are kept in during wetting and permanent immersion	Tap water; tap water saturated with lime (level doubled to obtain an orthogonal plane)

Table 2
Concrete mix design (kg/m^3)

Material	Siliceous aggregate (5/12)	Siliceous sand (0/5)	CEM I cement	Water
Amount (Kg/m^3)	1050	700	410	196.8

specimens, the second simulates heat treatment in a factory or heat generation within a massive member. The third consists of imposing wetting and drying cycles and the last is permanent immersion, during which length change of the specimens is measured. In some case the effect of the cycles was limited, especially when specimens are very reactive. In such cases, drying and wetting cycles may not be necessary to accelerate expansion in comparison with specimens which have not undergone those cycles. But, these cycles do not alter the long-term performance of the concrete and in some cases save time.

The proposed test is conducted on concrete specimens. This allows realistic experimental conditions to be used and limits alkali leaching.

2.2. Optimisation study

The objective was to estimate the impact of the various parameters involved in the test procedure, and then to specify experimental conditions which will allow the duration of the test to be minimized. The study was based on the application of a factorial experimental plan. The parameters studied are summarized in Table 1.

For curing time, drying temperature and the type of storage solution, the scope of the study was defined on the basis of knowledge derived from previous studies [12,27,28]. The temperatures of 38 °C and 60 °C were chosen so as not to provoke the dissolution of the ettringite during the test in view of the fact that the threshold values for the risk of DEF found in the literature are near 70 °C.

Lastly, the number of cycles, their duration and the length of time the specimens were kept in a wet enclosure were specified in order to cover a sufficiently large domain beyond which the time taken by the wetting and drying cycles is considered to exceed a reasonable duration for a test.

A "REX" strategy model proposed by Tagushi [31] was applied to the experimental plan, which had 18 combinations. The corresponding table is referred to as $L_{18}(2^1 \times 3^7)$ RNO-REX 1 and 2. This plan was repeated two times on concretes made with Portland cement CEM I and siliceous aggregate (Tables 2 and 3). After mixing and placing in cylindrical moulds (110 × 220 mm) the different concretes were subjected to the following heat treatment:

- The temperature was increased by 35 °C/h for 2 h;
- The temperature was maintained at 90 °C for 10 h;
- The temperature was gradually reduced from 90 °C to 20 °C over a period of 10 h.

This aggressive temperature cycle was chosen to ensure that DEF would be provoked in susceptible concretes in order to best evaluate the post curing conditions of the test. As described in part II of this study [30], the actual mix design and temperature cycle of the concrete are intended to be used in the test in practice.

The duration of curing in the wet enclosure (with a relative humidity of almost 100%) at 20 °C varied according to the factorial experimental plan.

Expansion was monitored using the low stiffness-free vibrating wire sensors developed by the LCPC [32]. These sensors were connected to an automatic data collection system. The error in the expansion measurements due to the apparatus is 0.0016% at a maximum frequency of 2000 Hz. The measurement range of this apparatus is of the order of 0.6% expansion.

2.3. Repeatability study

The repeatability of the test was quantified by applying the optimized test procedure to 10 concrete nominally identical specimens (Table 2) which had undergone the same heat treatment. For this study, the temperature plateau applied during the heat treatment was reduced from 90 °C to 80 °C. The expansion measurements were conducted using a Pfender ball extensometer distributed by Mohr and Federhaff [32]. The expansion curves given in Fig. 1 show the average expansion of the specimens obtained by monitoring three strain gauges for each specimen.

3. Results

3.1. Optimisation

Optimisation of the test was mainly concerned with the last two stages as phases 1 and 2 (mix design and heating cycle) are meant to represent the real conditions of concrete use and be

Table 3
Bogue computations of chemical and mineralogical composition of the CEM I Portland cement used (%)

SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	Cl^-	S^2
19.58	4.32	0.22	3.16	63.10	1.19	0.10	0.61	3.94	< 0.01	Néant
Insoluble residue	Loss on heating to 975 °C		MnO	Na_2O_{equi} [a]	C3S	C2S	C3A	C4AF		
1.21	1.33		0.06	0.5	55.2	14.3	6.1	9.6		

[a] $Na_2O_{equi} = Na_2O + 0.658 * K_2O$.

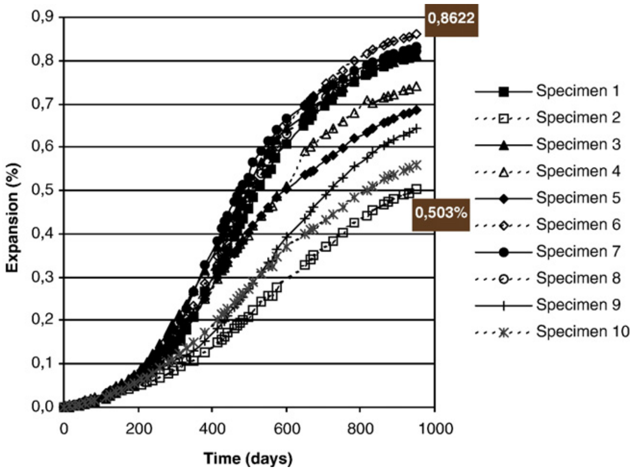

Fig. 1. Average longitudinal expansion — 10 concrete specimens (110×220 mm).

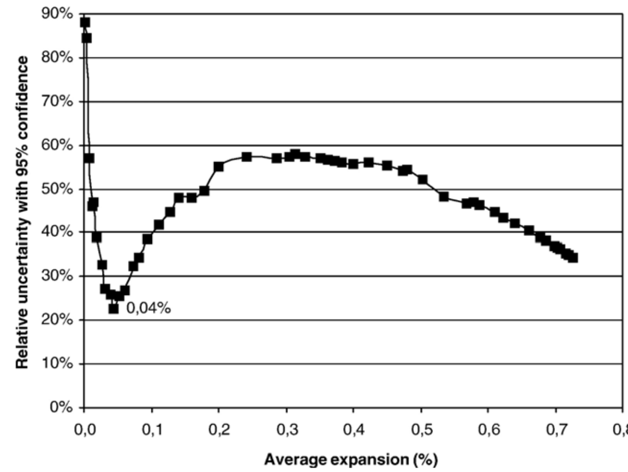

Fig. 2. Relative uncertainty according to average expansion.

adapted according to the concrete considered, as discussed in part II of this study [30].

The variance of the results was analyzed so that the role of each of the parameters could be studied. After immersion for 100 days, the various concrete specimens had expanded sufficiently for the impact of the parameters to be studied. The results are given in Table 4.

The experimental plan shows that the nature of the immersion solution (tap water or tap water saturated with lime) has no impact. This is probably due to the large size of our specimens. The plan also shows that increasing the duration of the curing phase which precedes the first drying cycle from 5 h to 28 days has no impact. The duration of a cycle and the number of cycles are the second and third order parameters. The largest effect on expansion was obtained for two cycles with a duration of 14 days. Inclusion of a period of storage at high relative humidity before the cycles was not considered to be significant as its effect was close to the natural variability of the system. This phase was therefore not included in the optimized test procedure adopted. The previous study [29] indicated that non-reactive concretes may be reactive when the drying phase is conducted at 60 °C. Therefore a temperature of 38 °C and a relative humidity of about 30% for the two drying phases were selected for the optimized test.

The optimized test procedure thus consists of two drying cycles (at 38 °C with a relative humidity of about 30%) and two wetting cycles (water at 20 °C) followed by monitoring the longitudinal expansion of the concrete specimens immersed in water at 20 °C. The test is designed to be used with a given mix design and temperature curing cycle representative of real concrete to allow investigation of "concrete mix design/heat treatment" pairs [33].

Table 4
Optimisation of performance test

Action	DoF[a]	S of squares[b]	Experimental variance	F_{exp}[c]	F_{crit}[d]	% Contribution
A	2	0.0027	0.0013	0.33	3.23	0
B	2	0.0278	0.0139	3.48	3.23	2
C	2	0.1509	0.0755	18.90	3.23	13
D	1	0.6628	0.6628	166.01	4.08	58
E	2	0.0624	0.0312	7.81	3.23	5
F	2	0.0006	0.0003	0.08	3.23	0
CD	2	0.1628	0.0814	20.38	3.23	14
Residue	32	0.0798	0.0040			7
Y	45	1.1498	0.0434			100

Analysis of variance. Results obtained after immersion for 100 days.

[a] DoF: Degrees of freedom;

[b] S of squares: sum of the squares of the difference with the overall average of the mean effect of each level of action;

[c] F_{exp}: ratio between the experimental variance of the action and the variance of the residues;

[d] F_{crit}: tabulated values (Fischer–Snedecor) of F below which the value of the action is not significant.

3.2. Repeatability

After 1000 days of testing, it was observed that the expansion of the 10 concrete specimens was quite variable (Fig. 1). The population of specimens has an average expansion of 0.75%±0.12%.

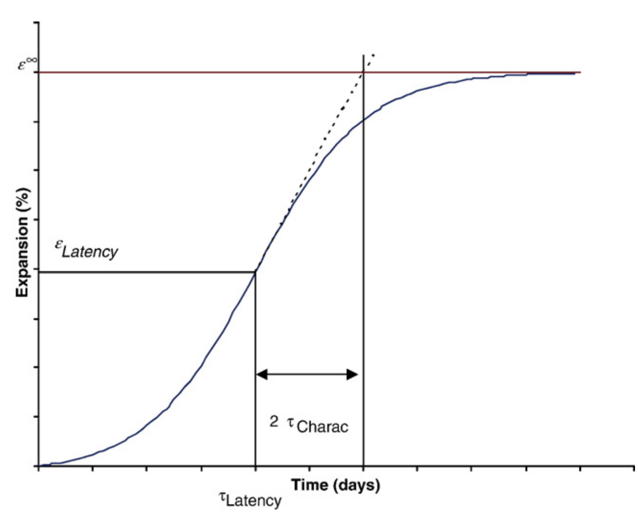

Fig. 3. Model expansion curve [32].

Table 5
Order of magnitude of the characteristic parameters of the average expansion plots for seven specimens that have undergone the performance test and a control specimen that has not been subjected to the wetting and drying cycles

	Latency period (days)	Characteristic time (days)	Final expansion (%)
Performance test	436	116	0.76
Specimen conserved in water at 20 °C	603	125	0.79

Fig. 2 shows the variation of confidence interval with a degree of expansion. At low expansions during the latency period, when the average expansion is less than 0.04%, the confidence interval is larger than the average expansion. As expansion starts the confidence interval is low, but increase due to different kinetics of expansion. At high levels of expansion, it falls with the level of expansion and reaches a value of 33% with a 95% confidence level (Fig. 2). These values are obtained for one heat cured concrete. A large study of the fidelity is actually conducted by several laboratories.

On the basis of these results, we were able to evaluate the impact of wetting and drying cycles on the expansion curves in comparison with the control specimens that have not been subjected to wetting and drying cycles. For this, three characteristic parameters (ϵ^{∞}, τ_{charac} and τ_{latency}) are determined to describe the expansion curves (Fig. 3) using the equation put forward by Larive [32], given below:

$$\varepsilon(t) = \varepsilon^{\infty} \frac{1 - e^{-\frac{t}{\tau_{\text{charac}}}}}{1 + e^{-\frac{t - \tau_{\text{latency}}}{\tau_{\text{charac}}}}}$$

where:

ϵ^{∞} is the maximum amplitude of expansion,

τ_{charac} is the characteristic time representative of half time of the period between the end of the latency period and the time when maximum amplitude of expansion is reach.

τ_{latency} is the latency time representative of the period when no expansion is measured.

The results are set out in Table 5. It is apparent that applying two wetting and drying cycles reduces the latency period by 5 months without significantly reducing or increasing the amplitude of expansion.

4. Discussion

The application of cycles after the heat treatment can reduce the latency period and consequently reduce the test duration. The impact of these cycles is probably twofold: on the one hand they may favour localized oversaturation of ettringite and on the other create and/or increase the size of microcracks in the concrete. The unstable propagation of pre-existing cracks requires less energy than the creation of cracks. The energy required for concrete expansion is, therefore, generated more rapidly when the concrete has been subjected to two heating cycles, which has an impact on the kinetics of expansion.

5. Conclusions and perspectives

The application of factorial experimental plan techniques has resulted in the optimisation of parameters in a performance test for evaluating the potential reactivity of "concrete mix design–heating cycle" pairs with regard to DEF. These tests were applied on one specific mix design. The results are validated with other mix designs and heating cycles in the second part of this paper.

The performance test proposed as a result of this research comprises four stages:

1. Concrete mix design and manufacture;
2. A heat treatment that simulates heat treatment in a factory or the heating that takes place in a massive cast in-situ concrete member;
3. Two cycles of wetting and drying. Each cycle lasting for 14 days and consist of the two phases: drying for 7 days in an enclosure at 38 °C with a relative humidity of 30% followed by immersion for 7 days in tap water at 20 °C.
4. Monitoring longitudinal expansion of the specimens immersed in water at 20 °C.

The nature of the immersion solution (water or saturated-lime solution) plays a secondary role. The concrete specimens should be sufficient size to avoid alkali leaching. The expansion mechanisms are not accelerated by imposing a large number of cycles or increasing the duration of the periods of wetting and drying. Likewise, raising the drying temperature from 38 °C to 60 °C does not lead to a marked acceleration in expansion.

Our statistical analysis has shown that the repeatability of this test is of the order of 33% with a 95% confidence. Additional tests are in progress to confirm these preliminary findings. The performance test must be validated before it can be used in the framework of a rigorous preventive approach. For this purpose, a study has been conducted on concrete that has been subjected to heat treatment representative of factory process or concrete heated within a massive cast in-situ member whose long-term performance in a wet environment is known from experience. The results of this study are presented in the second part of this paper [30].

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